

Measurement of Lead In Drinking Water

**Naval Command, Control and Ocean Surveillance
Center Research, Development, Test and Evaluation
Division
Code 36
San Diego, California**

July 19, 1996



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1. Introduction

1.1 Background Information

Lead leaching into drinking water from old piping systems poses a neurological threat to human health when consumed. Instances of facilities that do not comply with federal regulations on lead in drinking water occur in DoD and the private sector. Buildings have been removed from service, and bottled water brought into many others. Within the DoD, reversion of buildings to civilian use via the base closure process may require restoration of potable water systems.

Testing requirements will generate the need to conduct thousands of analyses at great cost in funding and time for DoD facilities over the next few years. The conventional method for identification of a problem typically entails collecting a predetermined number of samples, based on potential sources, followed by sample shipment to a certified laboratory for analysis. Turn-around time for results is typically two to four weeks and further testing, and delays, may be required if the source of contamination can not be determined with surety. An instrument that can perform rapid, in-field lead analyses will have a tremendous impact in reducing both the analytical costs and compliance time for all DoD facilities. This instrument would also provide the DoD and private sector with the capability to easily monitor remediation efforts on-site in a cost effective and timely manner.

1.2 Official DoD Requirement Statement

The measurement of lead in drinking water falls under component I.D. number A(1.1.a) Develop Improved Field Analytical Techniques under the Restoration/Site Characterization pillar; requirement number N2.V.1.p Assessment and control of lead in drinking water and requirement number N1.III.1.k Improved field analytical sensors, methods and protocols to supplement traditional sampling and laboratory analysis.

1.3 Objectives of the Demonstration

The objective of this demonstration is to provide performance, cost and field data for regulatory acceptance. To meet these objectives this project is divided into two major thrusts, field demonstration and laboratory validation. Validation of the instrument involves collecting data which will define the accuracy, precision and sensitivity of the instrument. Field demonstration will gather “actual” use data such as operator and expendable cost using the Metals Analyzer verses the traditional laboratory sample-and-ship method.

In this demonstration the portable automated Metals Analyzer will be used to measure, on-site, the drinking water lead levels in the building 100 complex at the San Diego Naval Submarine Base. These results will be compared with the standard graphite furnace atomic absorption spectrometry (GF-AA) and the inductively coupled plasma mass spectrometry (ICP-MS).

1.4 Regulatory Issues

A factor limiting the utilization and commercialization of electro-chemical methods for measurement of trace metals has been the lack of regulatory acceptance of this innovative technology. Acceptance of new methods is due to limited exposure, inadequate technical understanding, and lack of high quality data to support developer claims. Acceptance by the regulatory community requires good data, exposure of the technology and user demand.

1.5 Previous Testing of the Technology

Potentiometric Stripping Analysis (PSA) is capable of measuring over 40 different metals with enough sensitivity to detect concentrations in the low parts per billion range. It has been used to detect metals in such diverse fluids as drinking water¹, wine², sediment³, and blood⁴. An excellent summary of the technology can be found in *Stripping Analysis: Principles, Instrumentation and Applications*⁵.

2. Technology Description

2.1 Description

A prototype automated laboratory lead analyzer was developed which is based on the electrochemical technique of PSA pioneered by D. Jagner in the early 70's⁶. Recent

advances in electronics now enable the technique to perform completely automated lead analyses to 1 part per billion (ppb) in about five minutes. The system consists of (1) a computer; (2) custom control and data acquisition and analysis software; (3) a custom computer controlled potentiostat and data acquisition circuitry; and (4) a custom flow-through electrochemical cell module and sample handling components.

The instrument will provide a capability to perform on-site, discrete analyses or automatic, continuous monitoring at programmed intervals and is designed for use by non-technical personnel. This capability will permit near-real-time tracking of lead contamination throughout a distribution system, expanded monitoring in support of remediation efforts, and lead measurements where sources must be frequently monitored over extended periods.

The high sensitivity of PSA can be attributed to the unique coupling effect of effective preconcentration steps and advanced measurement procedures. The electrode consists of a glassy carbon rod on which is deposited a thin mercury film. The electrode is exposed to the sample and a negative voltage is applied (Figure 1) the lead is reduced and forms an amalgam on the electrode. This concentration phase can last from 1 to 120 seconds depending on the amount of lead in solution. Typically for lead concentrations in the range of 1-30 $\mu\text{g/L}$ a 30 second plating time is sufficient. After the concentration phase the voltage is removed and the potential of the electrode is measured.

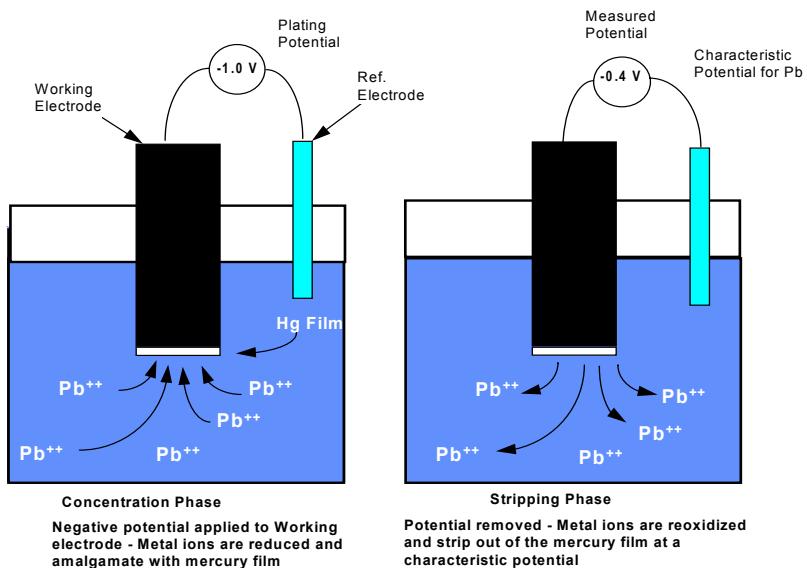


Figure 1. Potentiometric Stripping of Lead

The potential drops until it reaches the characteristic value for lead oxidation. At this voltage the lead is oxidized and stripped out of the mercury film. The potential remains constant until all the lead is oxidized producing a plateau in the voltage versus time graph (Figure 2). The width of the plateau is proportional to the concentration of lead originally in solution.

Potentiometric Stripping Curve

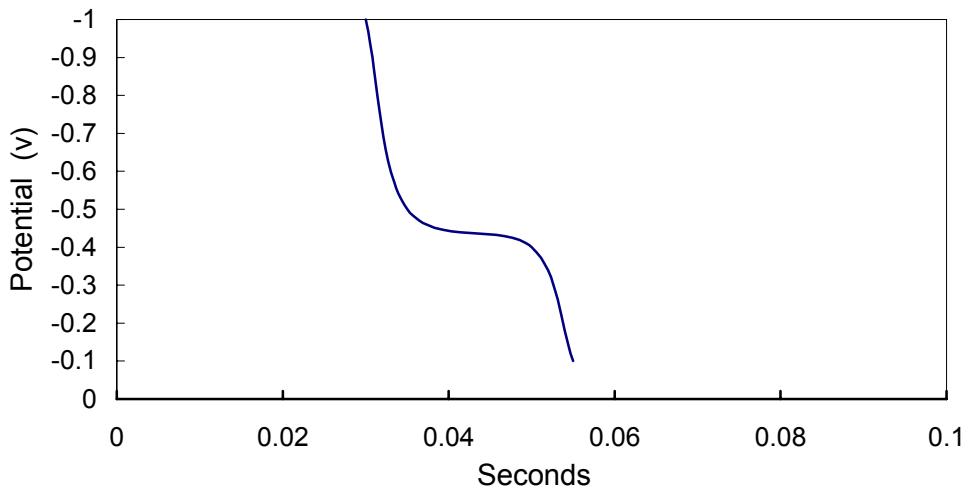


Figure 2. A potentiometric Stripping Curve. While the lead is oxidizing and stripping out of the mercury film the potential remains constant. The width of this plateau is proportional to the original concentration of lead in solution.

To calculate the true concentration of the metal a standard curve is generated by adding known quantities of lead to the test sample and repeating the measurement. This entire process is totally automated and proceeds without any intervention. When the concentration of lead has been determined (usually in less than 3 minutes) the instrument displays and logs the concentration and the relevant measurement statistics, pumps out the test sample, rinses the cell, and waits for either an operator command, external signal or the next programmed sampling interval.

2.2 Strengths, Advantages and Weaknesses

The strengths of PSA are accuracy, sensitivity, versatility, and simplicity. Increasing the concentration phase of PSA allows the instrument to measure lead concentrations in the single digit ng/L range although at these low levels sample handling becomes such a dominate factor that field use becomes impractical. The instrument is very versatile allowing the user to operate the instrument in single sample mode, multiple runs on a single sample, or totally automated sample and measure mode at programmed intervals for up to ten days.

As will all trace metal measurement techniques the major weakness is in matrix effects interfering with the measurement. Interference for lead measurements include high concentrations of organics, solids, or salts. The levels at which these begin to interfere are well above standards set by the Safe Drinking Water Act. Concentrations of thallium(I) above 100 μ g/L have also been found to interfere with the measurement of samples containing the 10 μ g/L of lead.

2.3 Factors Influencing Cost and Performance

Factors influencing cost and performance include capital equipment cost, operator time and the cost of consumables. By far the dominating factor is operator time. There are two ways to approach reducing operator cost, lowering the hourly rate of the operator and reducing the time required per sample. The instrument was developed with an easy to use windows interface and automated self diagnostics allowing the use of less expensive technicians to operate the instrument when compared to the traditional GF-AA instrument. The Metals Analyzer is totally automated once the sample is introduced allowing a short measuring time of less than three minutes. Due to the small sample volumes required (<3ml) the amount of reagents is kept to a minimum.

3. Site/Facility Description

3.1 Background

There were four criteria used for site selection, they were:

1. Availability of potable water and electrical power.
2. A minimum of 10 sampling sources
3. The ability to shut off all faucets for 24 hours before measurement (As per EPA sampling protocol).
4. Eight hours of unrestricted access to the facilities during testing

The demonstration site is located at the San Diego Naval Submarine Base. The area tested was the building 100 complex. (see Appendix C) This complex was initially built in 1927 as a naval gun battery. It has since been converted to general office use. The complex also includes other mixed use buildings containing offices and storage, and a warehouse facility, that was constructed in 1988.

3.2 Site Facility Characteristics

The age of the drinking water plumbing system ranged from 1927 copper pipe soldered with high content lead solder to 1988 copper piping soldered with lead free solder. The faucets tested included interior and exterior faucets, refrigerated water fountains and two non potable water faucets whose water source was unknown.

4. Demonstration Approach

4.1 Performance Objectives

The contaminant measured by the analyzer in this demonstration was lead ions in drinking water down to a level of 5ppb. The data when compared with GF-AA and ICP-MS should have a correlation coefficient of 0.95. The instrument generates process waste containing a dilute solution of acid, lead, and mercury. A single run will produce a 2.7 ml solution with a pH of 2 and containing approximately 300 ppb mercuric chloride and 33 ppb lead. From setup, sample collection to final result should take less than 15 minutes.

4.2 Physical Setup and Operation

The instrument requires little in terms of infrastructure. It requires a 110v electrical outlet and approximately 10 sq. ft. of table space. The hazardous waste generated by the instrument (approximately 2.5 ml per run) is automatically collected in a waste container. For this demonstration two individuals were used, a sample collector and an instrument operator. The demonstration was conducted on a Saturday starting at 0900 and concluded that evening at 1800.

Sampling Procedures

The portable lead analyzer was carried to the building being tested (see Appendix C for sample locations). The instrument was operated in a convenient location inside the building. The building samples were collected and split. Each sample was clearly labeled as to the location of its source, the date and time it was collected, and the name of the collector. One-half of the sample was analyzed by two independent water quality testing laboratories. The other half was analyzed by the Metals Analyzer.

The samples that were sent to the laboratories were acidified to a pH of 2.0 using nitric acid. The remaining sample was acidified to a pH of 2.5 and brought to the instrument for analysis. The analyzer processed the samples and the data was stored on disk. These results were compared with the results from the testing laboratories.

Analytical Procedures

The samples were analyzed on site using PSA, splits of the sample were preserved and analyzed using GF-AA and ICP-MS. Specifics pertaining to sampling and analysis can be found in the “Demonstration Plan for the Automated Metals Analyzer”.

5. Performance Assessment

5.1 Performance Data

Fourteen samples were collected at the sites shown on the map in Appendix C. Each of these samples were split and preserved with acid, the PSA aliquot was measured on-site and the GF-AA, and ICP-MS aliquots were sent to independent testing laboratories. Figure 3 is a comparison between the results obtained by ICP-MS and GF-AA. Ideally the results would lie along a 45° line crossing at the zero axis with a correlation coefficient of 1.0. For each sample, the GF-AA instrument's values were lower than the values obtained using the ICP-MS instrument. However, a correlation coefficient (R^2) of 0.99 indicates that this discrepancy is probably due to differences in initial calibration rather than an inherent instrument error. Comparing GF-AA values with PSA (Figure 4) gives similar results, with the GF-AA generally measuring values lower than PSA again with a high correlation coefficient. Similarly in Figure 5 the ICP-MS results are generally higher than PSA. Looking at all three measurements together in Figure 6 it can be seen that where large discrepancies between ICP-MS and GF-AA exist the PSA measurements always fall between.

These results indicate that within experimental error field measurement using PSA will give equivalent results when compared with traditional methods. It has been our experience that the discrepancies between the GF-AA and ICP-MS laboratory results are not unusual when conducting comparisons between laboratories. What should be emphasized is the excellent linearity (>0.97) between all three methods. More consistent calibration protocols between the laboratories would likely produce lead levels with less spread.

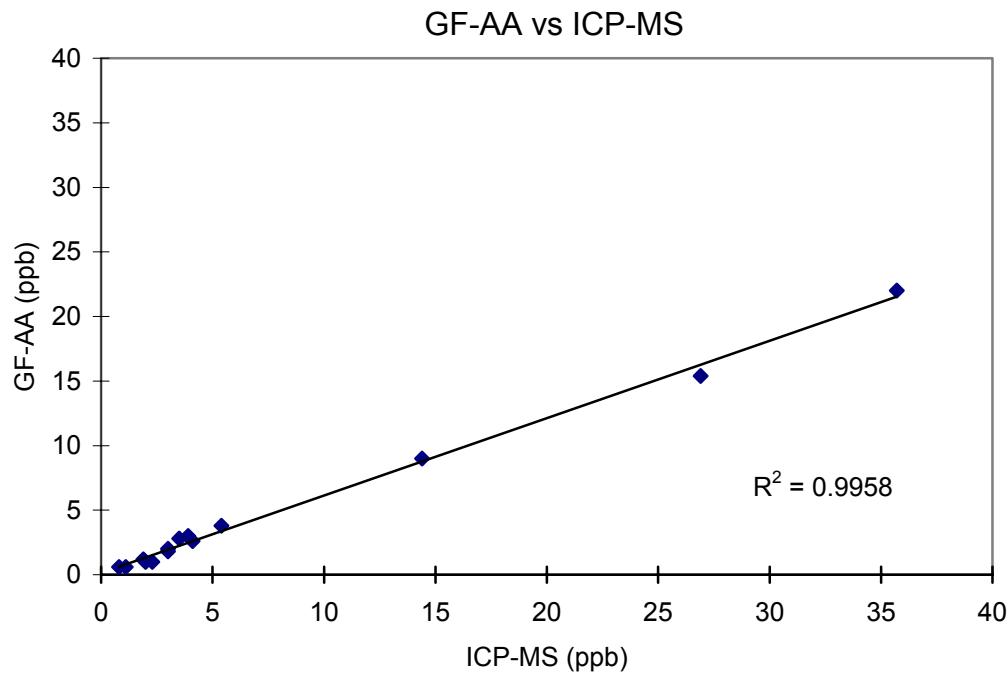


Figure 3 A comparison of split sample results between graphite furnace atomic absorption spectrometry and inductively coupled plasma mass spectrometry. Each data point represents one sample, the X value is the amount of lead measured by the ICP-MS and the Y axis is the amount of lead measured by the GF-AA.

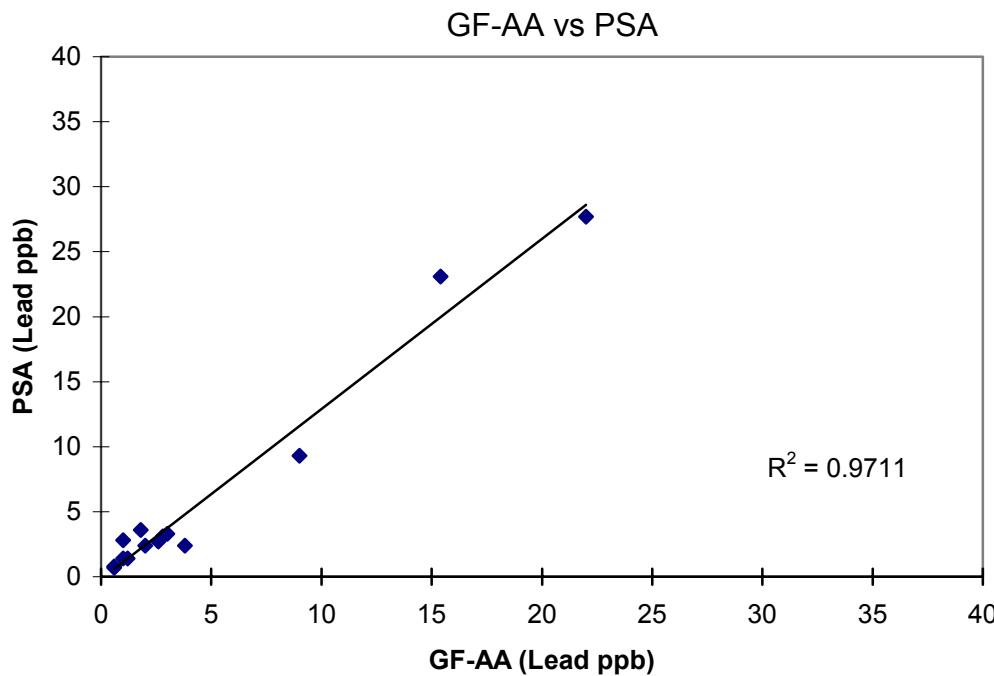


Figure 4 A comparison of split sample results between graphite furnace atomic absorption spectrometry and potentiometric stripping analysis. Each data point represents one sample, the X value is the amount of lead measured by the GF-AA and the Y axis is the amount of lead measured by PSA.

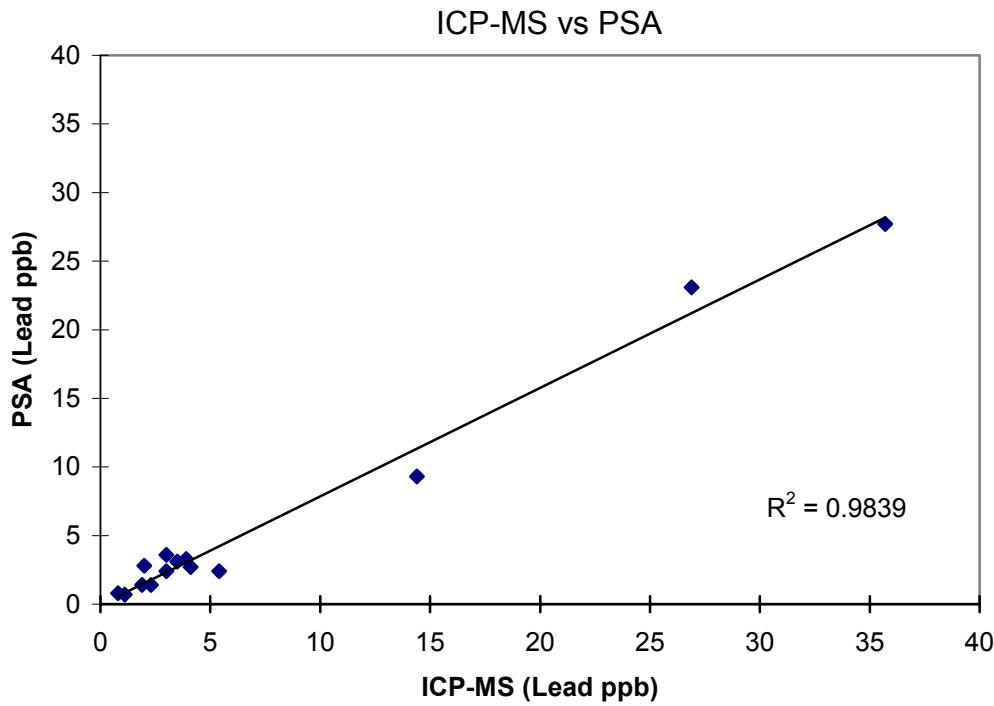


Figure 5 A comparison of split sample results between inductively coupled plasma mass spectrometry and potentiometric stripping analysis. Each data point represents one sample, the X value is the amount of lead measured by the ICP-MS and the Y axis is the amount of lead measured by PSA.

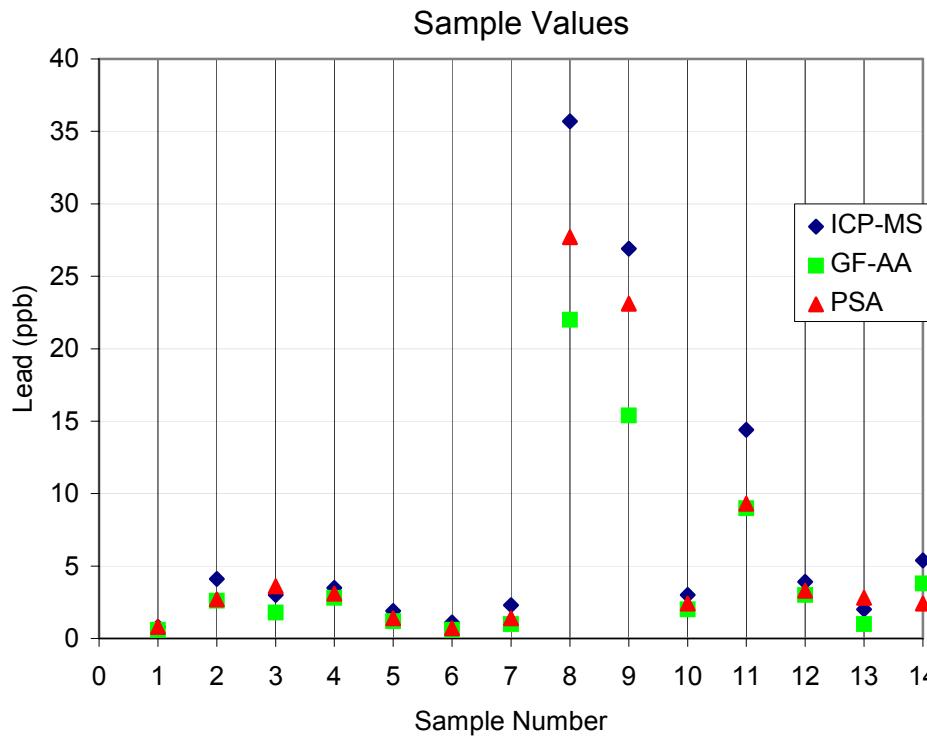


Figure 6 The lead levels for each sample measured by inductively coupled plasma mass spectrometry, graphite furnace atomic absorption spectrometry, and potentiometric stripping analysis.

5.2 Data Assessment

A series of laboratory tests were conducted to assess the performance of PSA in measuring lead in drinking water. The water was collected from various sources within the Las Cruces New Mexico, Water Utility District which uses ground water as a source. In Figure 7 a single sample was measured nineteen times, the relative standard deviation of all measurements was less than 3.2%. Figure 8 is a correlation between PSA and GF-AA of a single sample spiked to 15 different levels of lead. This analysis resulted in a correlation between PSA and GF-AA of 95%. The linearity of the instrument range was tested from 0 to 300 ppb lead concentrations. To avoid biasing the results with the data manipulation software built-in to the instrument the actual analog to digital samples were collected, the voltages that were within the characteristic stripping potential (see Figure 2) were summed and that value was plotted for each of the concentrations measured. Within the 0-300 ppb range a correlation of 99% was calculated indicating excellent linearity.

An interference study was conducted with 15 different metal ions. The water sample was spiked to a 10 ppb lead level and split three ways for each of the interfering metals tested. One aliquot was spiked to 50 ppb with the interfering ion and the other was spiked to 100 ppb. The results are shown in Table 1. Only Thallium(I) showed a marked change at 50 and 100 ppb concentrations. Indium(III) and Antimony(III) also showed a slight interference effect (above 10%) at both concentrations.

PSA compares very favorably to current standard methods with excellent linearity up to 300 ppb. Interferences with other metals likely to be found in drinking water are minimal. This data indicates that PSA is an appropriate method for measurement of lead in drinking water.

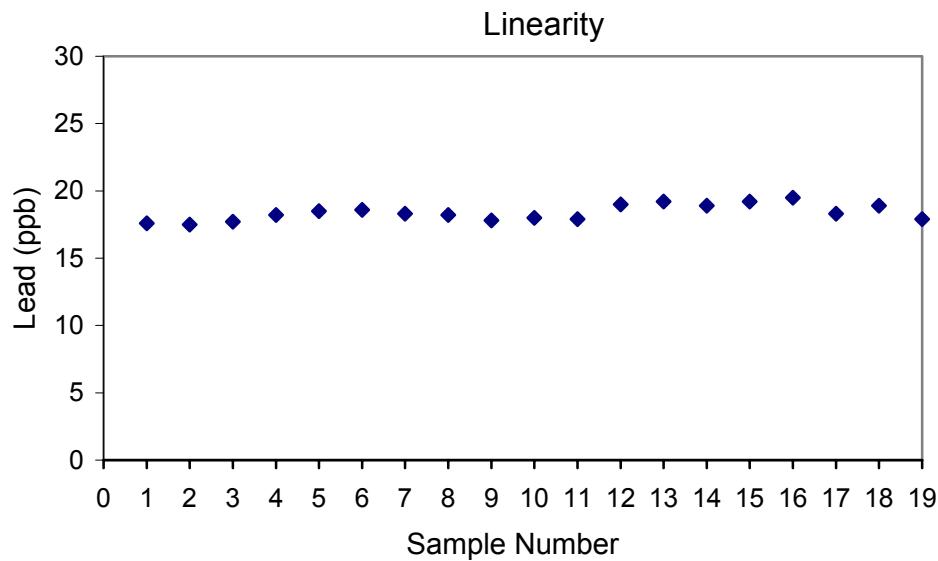


Figure 7. A single sample of drinking water measured nineteen times. The relative standard deviation for this data set is 3.17%.

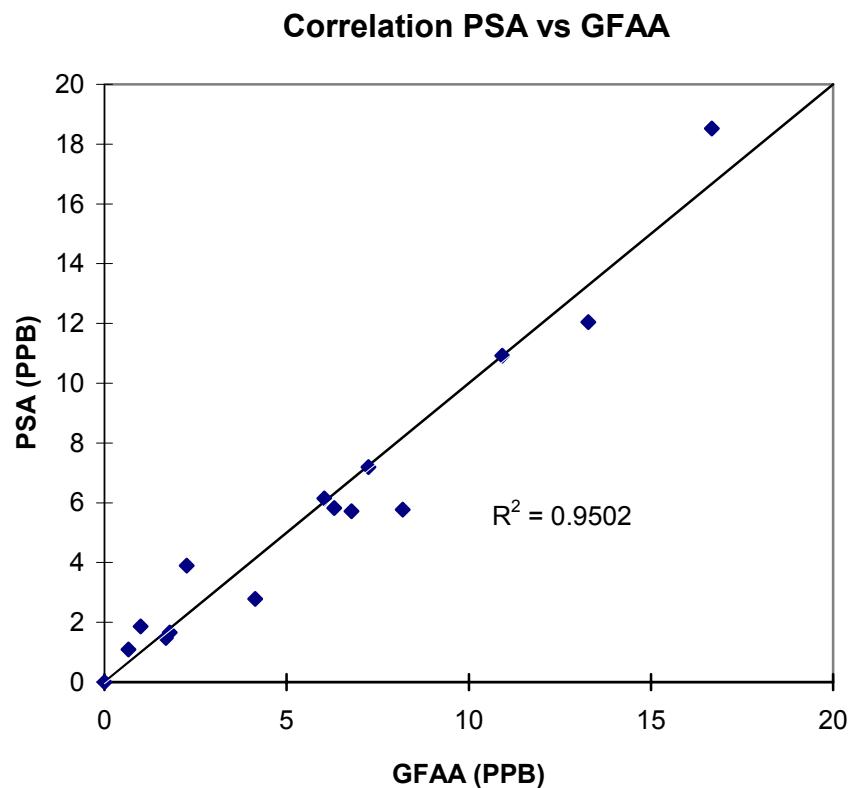


Figure 8. Correlation of drinking water samples spiked to different levels of lead.

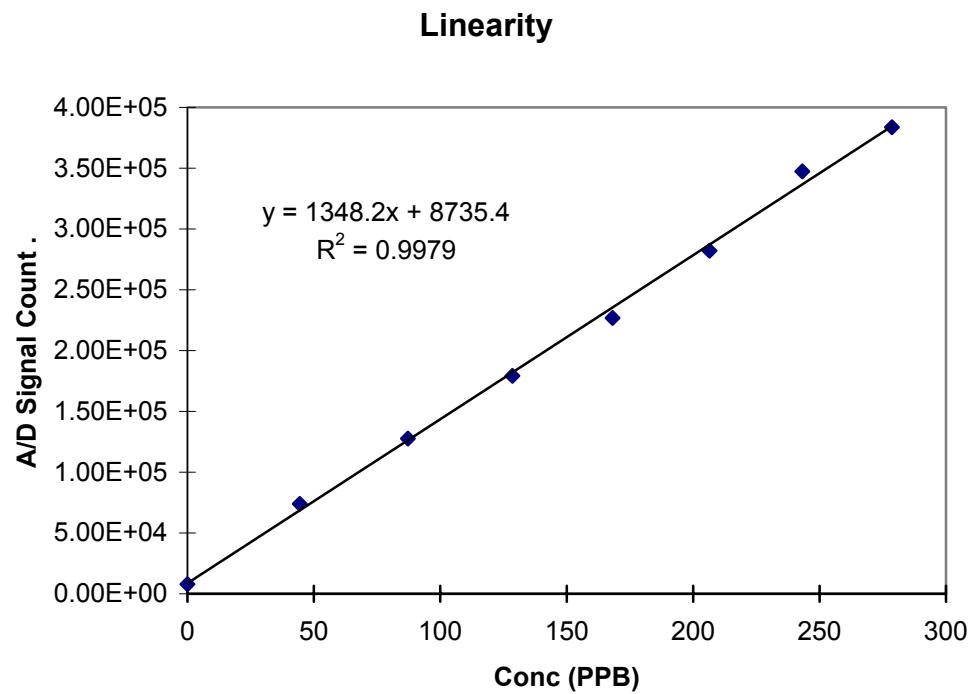


Figure 9. Linearity of Metals Analyzer over a range of 0-300 ppb. The actual number of analog to digital converter bins which were at the characteristic stripping voltage were counted for each addition of lead added to the sample.

Table 1. Interference study of 15 metals at 50 and 100 ppb concentrations. The original sample was spiked to 10 ppb lead and split three ways for each metal tested. Aliquots two and three were spiked, with the interfering ion, to 50 and 100ppb respectively. The percent change in measured lead levels between the spiked samples and the original 10ppb lead sample was recorded.

Interfering Ion	Interferences			Interference Status	
	50 PPB		100 PPB		
	Interfering Ion Added	%Change	Interfering Ion Added	%Change	
Aluminum(III)		7.75		5.18	No
Antimony(III)		16.20		15.03	Yes
Arsenic(III)		5.22		-11.37	No
Bismuth(III)		5.76		3.24	No
Cadmium(II)		3.87		11.38	No
Calcium(II)		-5.70		-7.26	No
Copper(II)		0.87		13.74	No
Chromium(VI)		8.69		6.16	No
Indium(III)		10.94		-15.04	Yes
Iron(III)		7.14		7.41	No
Manganese(II)		0.71		11.79	No
Nickel(II)		-10.20		6.01	No
Thallium(I)		-38.35		-45.05	Yes
Tin(IV)		8.69		15.19	No
Zinc(II)		7.36		11.85	No

5.3 Technology Comparison

For drinking water PSA compares very favorably to ICP-MS and GF-AA. Correlation of data (Figures 4, 5, and 8) always results coefficients above 0.95. The Metals Analyzer has several advantages over ICP-MS and GF-AA instruments. The Metals Analyzer is portable and capable of unattended field operation and requires a minimal technical level to operate. In contrast GF-AA and ICP-MS instruments are large laboratory instruments requiring a very high level of expertise and substantial infrastructure to support plus they are not well suited to automated long term monitoring.

6. Cost Assessment

6.1 Cost Performance

Table 2 is an estimate of the costs incurred over an expected 10 year life span of the instrument assuming a processing rate of 100 samples per month. The labor rate was fixed at \$50 per hour resulting in a total life cycle cost of \$207K.

Table 2 Cost performance table for the Metals Analyzer over a 10 year life span.

Cost Performance					
Cost by Category					
Start-up		Operation & Maintenance		Demobilization	
Activity	\$K	Activity	\$K	Activity	\$K
Capital Equipment	25.0	Labor	150.0	Disposal (just throw it away)	0.0
		Consumables & supplies	18.0		
		Effluent disposal	12.0		
		Training required to operate equipment	2.0		

6.2 Cost Comparisons to Conventional and Other Technologies

Costs are best compared with current practice for determining lead levels in drinking water. Typically samples are collected and preserved by acidifying the sample with nitric acid, the samples are packed in ice and shipped over-night to a trace metal laboratory. Cost for the laboratory analysis can vary from \$20 to \$75 per sample depending on the laboratory and the volume of samples. Even at the lower \$20 per sample PSA compares very favorably at less than half the cost of conventional methods. Typically the turnaround time for conventional methods is 2-4 weeks verses 15 minutes with PSA, this allows immediate resampling if results warrant it. Using traditional methods resampling time could take over a month.

Table 3 Cost breakdown per sample between traditional laboratory GF-AA analysis and on site PSA analysis

Cost Breakdown Per Sample			
GF-AA		PSA	
Activity	Cost	Activity	Cost
Sample Collection (\$50/hr) - 5 min collection - 10 min preservation, labeling packing - shipping	4.17 8.34 1.86	Sample Collection and Analysis (\$50/hr.) - 5 min collection time - 10 min operator time	4.17 8.34
Sample Analysis (contract with laboratory)	50.00	Consumables - Reagents - Electrodes	1.25 0.25
		Capital Cost of Instrument - \$25k/40,000 samples	0.63
		Haz/Mat Disposal (3ml)	1.00
Total	64.37		15.64

7. Regulatory Issues

7.1 Approach to Regulatory Compliance and Acceptance

EPA Cincinnati has developed a protocol for nationwide approval of new or revised methods for inorganic analyses in national primary drinking water regulations monitoring. They were contacted early in the project. EPA's estimate of approval of a new standard was \$375K requiring 3 years for final acceptance. Based on this it was realized that funding from ESTCP alone would not be enough for full acceptance. Discussions with the programs CRADA partner ensued. It was agreed that they will pursue regulatory acceptance using data provide by this project. This approach was discussed at the ESTCP IPR and is being pursued. Due to the high costs and lengthy approval process EPA is now developing a streamlined approval of analytical method protocol in response to the current Administration's Environmental Technology Initiative. The goal of the proposed protocol is to promote the use of innovative techniques while ensuring equivalency with approved method. This holds promise of a much more rapid and less expensive approval of PSA for drinking water in the near future.

Currently we are pursuing regulatory acceptance outside of ESTCP by incorporating the instrument into monitoring requirements stipulated in National Pollution Discharge Elimination System (NPDES) permits. We are working with Norfolk Naval Ship Yard to incorporate use of the instrument to monitor trace metal for NPDES. Discussions with Puget Sound Naval Shipyard are underway for the use of the instrument for their NPDES monitoring requirements.

8. Technology Implementation

8.1 DoD Need

Over 250,000 violations of the Safe Drinking Water Act (SDWA) occurred during 1991 and 1992. Over 300,000 occurred in 1993 and 1994. 43% of community water systems had violations including 15,000 episodic or chronic violations of National Primary Drinking Water Standards (NPDWS). Non-community water systems average over 10,000 NPDWS violations annually. DoD has over 275 water systems which fall under SDWA, NPDWS and/or OPNAVINST 5090.1B. 5090.1B requires the control and elimination of the dangers of lead exposure from drinking water by monitoring of priority areas, monitoring of water coolers and lead and copper rule monitoring.

8.2 Transition

The Trace Metals Analyzer is presently licensed commercially and is scheduled for production in 1997, it will be made available through GSA contract. Transition of PSA technology and ongoing developments is subject to a Cooperative Research and Development Agreement through 1997. NRaD maintains a laboratory, portable and a survey ship instrument for demonstration. Two other instruments have been transitioned to the Naval Facilities Engineering Service Center (NFESC) for use by Public Works Centers and Environmental Field Divisions through the NFESC transition coordinator.

9. Lessons Learned

As regulatory certification proceeds it has become apparent that the initial cost estimate and time table were wildly optimistic. This initial estimate was base on literature published by the EPA. The most important lesson learned is not to trust the literature. Talking directly with the regulators before beginning any regulatory acceptance is a must. This is not as easy as it sounds. The acceptance path is not clearly laid out and is subject to the latitude given regulatory personnel by the agency. Different and sometimes conflicting answer will come from different sources. The climate for

acceptance can vary from down right hostile, “we will not accept any new method that produces hazardous waste”, to mildly enthusiastic “electro-chemical methods could potentially revolutionize trace metal analysis”.

10. References

¹Jagner, D., et. al., Analytical Chemistry, 1993, **278**, 237.

²Jagner, D., Renman, L., and Wang, Y., Electroanalysis, 1993, **5**, 283.

³Madsen, P., Drabaek, I., and Sorensen, J., Anal. Chim. Acta., 1983, **151**, 479.

⁴Jagner, D., Analytical Chemistry, 1978, **50**, 13, 1924.

⁵ Wang, J., *Stripping Analysis: Principles, Instrumentation and Applications*, VCH, New York, 1985.

⁶ Jagner, D., Analytical Chemistry, 1978, **50**, 13, 1924.

Appendix A

Points of Contact

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Appendix B

Data Archiving and Demonstration Plan

Field validation data collected as part of this project is archived in the NRaD Environmental Sciences Division Program Office. Electronic copy of the field data is stored on a 3.5" floppy disk in the data acquisition software custom binary format. Hardcopy plots laboratory results, data reports, and test plan are also maintained in the Program Office. Requests for copies of the data or reports should be made to Mr. Mike Putnam at the address listed in Appendix A.

Appendix C

Site Maps

The following are site maps for the field demonstration. The demonstration was conducted at the San Diego Naval Submarine Base located at the southern end of Point Loma 3 miles west of San Diego CA.

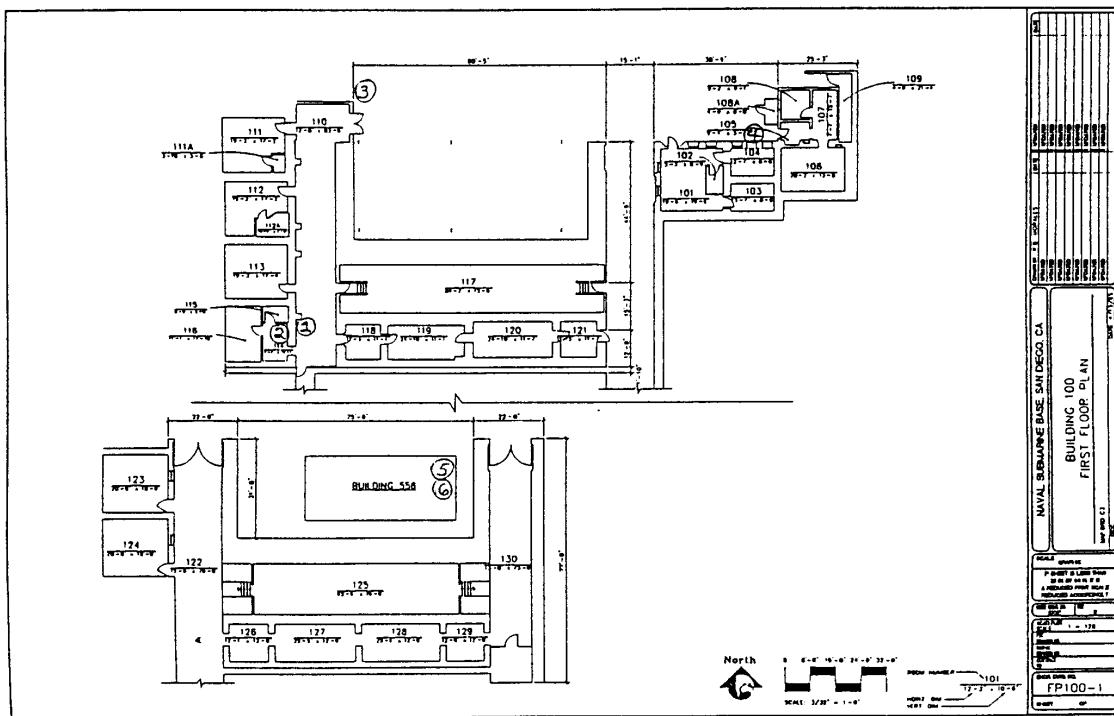


Figure C 1 Location of sites 1-6

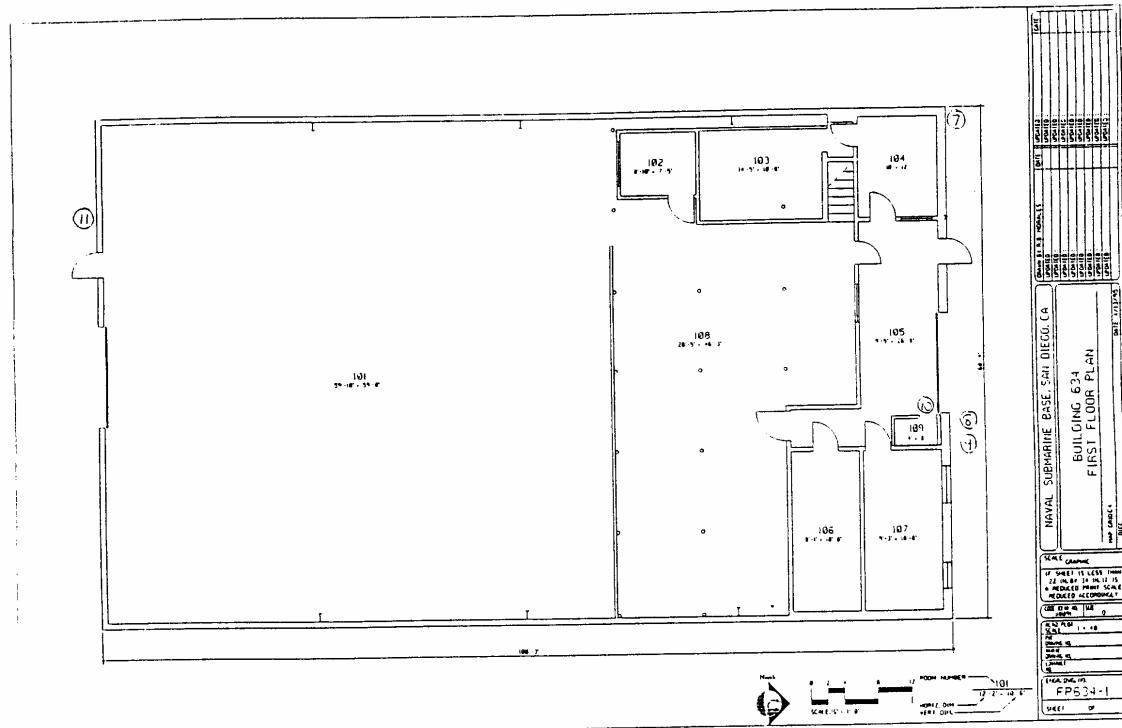


Figure C 2 Location of sites 7-11

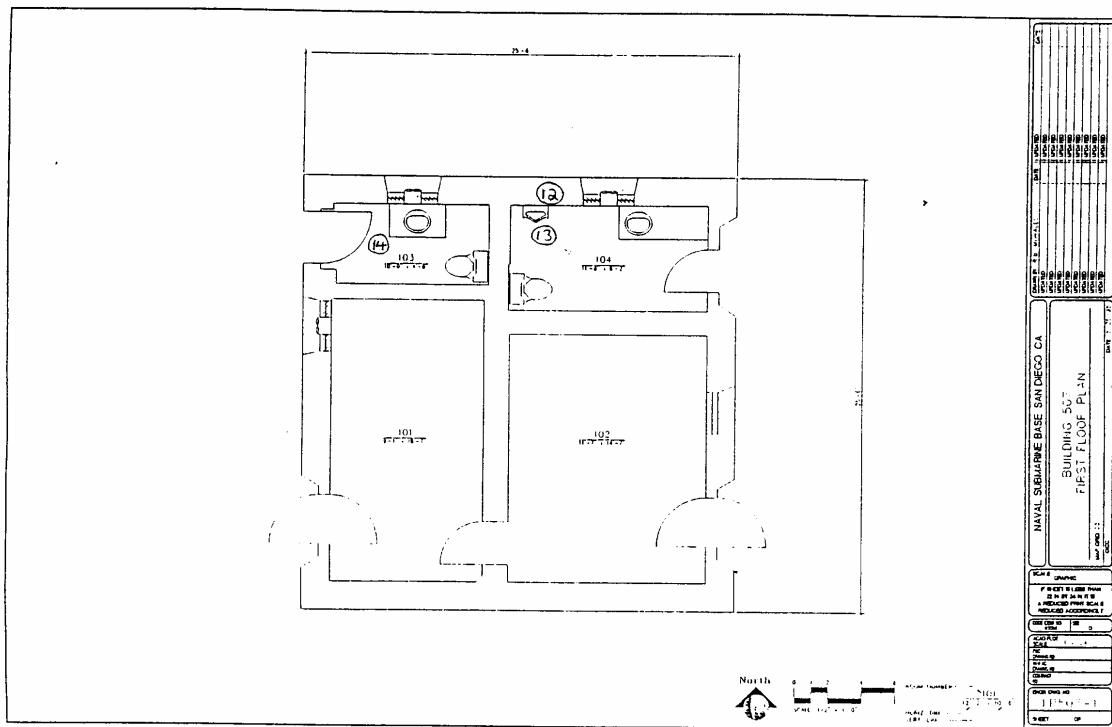


Figure C 3 Location of sites 12-14

Appendix D

User's Manual

The following user's manual was developed with funding from ESTCP. It is a comprehensive guide on the features, use and maintenance of the Metals Analyzer.